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METHOD OF MANUFACTURING SILICEOUS INSULATING MATERIAL

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This invention relates to the manufacture of light weight siliceous materials. More particularly, the invention relates to new types of heat insulating materials based, in the main, upon silica in an improved state with respect to its physical and chemical properties.

The manufacture of high temperature heat insulation has been more or less restricted to the utilization of siliceous materials due to the fact that magnesia types of insulation disintegrate rapidly or check severely at temperatures upwards of 1000° F. For this reason, the industry has relied extensively upon the use of various forms of silica which are reacted with binders, for example those of the calcareous type such as lime, Portland cement, shale, etc. The practice of manufacturing such insulation is quite old in the art as evidenced by a patent issued to Brown, U. S. 311,287. The technique of preparing such insulation from diatomaceous earth and calcareous binders has been somewhat improved, the main emphasis being laid upon the preparation of insulating blocks or slabs of low density and high mechanical strength. These objects have, in part, been achieved by adding asbestos fiber to the diatomaceous earth-calcareous mixture. It has also been proposed to add certain colloids or dispersing agents to an aqueous slurry of the reactants in order to effect improved stability of the reaction mixture in the period during which the diatomaceous earth reacts with the binder to form a pre-hardened body. It has been possible to produce siliceous insulation blocks or slabs having a density of about 12 to 30 lbs./cu. ft. with a modulus of rupture between about 30 and 150 lbs./sq. in., but this is only achieved by employing highly specialized forms of asbestos fiber.

The preliminary reaction between diatomaceous earth or other siliceous materials and a calcareous binder is usually carried out in such a manner that the reactive mix will not "set up" or harden before the mixture is placed in molds of suitable shape and design. Following the pre-set or hardening, the resulting siliceous bodies are then heated, usually by means of an autoclave, at temperatures in excess of 100° C. to further accelerate and increase the extent of the reaction between the siliceous material and the binder. It is the usual practice to preliminarily react the siliceous material and binder in the presence of colloids or agents for suspending the reactants for a period of time approximating two hours at temperatures of the order of 80° to 100° C. Following this preliminary reaction whereby the materials set to a pre-determined shape, the aforementioned indurating process is conducted to produce the final product. A desired improvement upon this process would substantially reduce the time required to preliminarily react the siliceous material and binder or accelerate the pre-setting time and which would also reduce the weight of the block without any substantial reduction in strength.

It is a further object of the invention to provide a process for the manufacture of improved siliceous materials from minerals such as diatomaceous earth, silica flour, and other siliceous materials which are reactive with alkaline earth compounds.

It is still a further object of the invention to provide a process for the manufacture of siliceous refractory insulating materials from minerals such as diatomaceous earth, silica flour, and the like together with calcareous binders in order to substantially reduce the pre-set time formerly required in such manufactures.

The above objects as well as others which will become apparent upon a complete understanding of the invention as hereinafter fully described, are accomplished by reacting a composition comprising finely divided siliceous material with an alkaline earth silicate-producing compound reactive with said material, acidifying the resulting product with an acid which will give a soluble alkaline earth compound while maintaining the pH of the mixture between about 3.0 and about 8.0. The acidification step is followed by recovering the siliceous product in the manner hereinafter described. The siliceous product is then admixed with a binder, preferably in an aqueous slurry, after which the latter is heated at a temperature and pressure sufficient to solidify the slurry while minimizing substantial evolution of water therefrom. The resulting solidified material can be used directly as a low-temperature refractory. Alternatively, it may be heated to above 100° C. to substantially remove all of the occluded water. In some instances, it is desirable to fire the refractory to temperatures up to 500° C. to enhance its properties.

In a broad embodiment of the invention, we react a finely divided silica-containing material of the type more particularly subsequently described herein with an alkaline earth silicate-producing compound, preferably a compound of lime which is reactive with silica and certain other siliceous materials, at a temperature and for a time sufficient to cause the formation of hydrous alkaline earth metal silicate upon the exterior surface and in the interstices of the siliceous material. The resulting composition is then acidified with an acid which will react with the hydrous alkaline earth silicate to produce a compound or salt which is soluble either in the acidified mixture or in water or in other suitable solvent. The pH of the reaction mixture is maintained between about 3.0 and about 8.0 during the acidification to insure reaction of the hydrous alkaline earth silicate with the acid agent. This results in the formation of a siliceous aggregate having a base substantially that of the starting material, and which is further characterized by a siliceous coating integrally bonded to the surface and interior surfaces of each particle. This renders the aggregate highly reactive, particularly towards calcareous binders of the type employed in the manufacture of our siliceous-calcareous high-temperature insulation.

Our improved siliceous products are to be distinguished from certain silicic acid-aggregate compositions consisting of silicic acid which has been precipitated from a solution of a water soluble alkali metal silicate by means of a mineral acid in the presence of a mineral aggregate such as rock, shale, diatomaceous earth, etc. Such products are characterized by a loose bonding of the silicic acid to the aggregate and the resulting coating does not form an integral and fixed part of the particle.

Our novel siliceous product is then mixed with a binder which will be essentially inorganic in nature in the event that the final siliceous material is to be used as a high-temperature refractory. We prefer to employ binders reactive with the silica such as the oxides, hydroxides, carbonates of alkaline earth metals especially calcium, magnesium, and barium. We may also employ binders such as water glass, clays, etc. as well as organic binders such as asphalt, phenol-formaldehyde resins, urea-formaldehyde resins and other polymeric materials. To improve the strength of the final product, we prefer to have present a fibrous material which may be organic in nature but which is preferably of mineral origin, particularly if the fiber is to withstand severe high-temperature conditions in subsequent utilization of the final product. The mixture of binder, fiber, and our improved siliceous materials together with sufficient water to give a workable or pourable mixture is placed in molds or dies or other suitable forming media, the forming means and slurry being heated at elevated temperatures and pressures to "pre-set" the mixture. Due to the characteristics of our improved siliceous products, we have found that the mixtures will set considerably faster than mixtures heretofore employed in the manufacture of siliceous refractory materials, thereby substantially reducing the time of the overall operation. Following the pre-set stage, the solidified forms or shapes are indurated by heating at elevated temperatures and pres-